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MEMORANDUM

DATE. 28 December 1998

TO· David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle
RMM Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data
Laboratory Batch: K9806066
Site: Duwamish River

WORK ASSIGNMENT NO 46-35-0JZZ

WORK ORDER NO.: 4000-019-038-5200-00

DOC. CONTROL NO.: 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U S. EPA, Region X
Dena Hughes, Site Manager, WESTON, Seattle (memo only)
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of four sediment samples, laboratory batch K9806066, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. The sampls were analyzed by gas chromatography with an FPD detector. The samples were numbered.

98364033 98364034 98364035 98364036

Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240 1, February 1994), modified to include specific requirements of analytical methods

1. Timeliness

Holding time limits of 7 days for sample extraction and additional 7 days for analysis were established in the project Sampling and Analysis plan. All samples were extracted 55 days after sample collection, exceeding the 7 day holding time criterion. However, prior to extraction, samples were stored frozen, thus extending the holding time. Samples were extracted within the 12 month holding time recommended by PSEP for frozen samples.

2 Detection Limits

Instrument detection limits met project required quantitation limits with the following exceptions

Sample	Compound	QL Goal (µg/Kg)[1]	Reported QL (µg/Kg)
98364033	Tetra-n-butyltin	10	15
98364034	Tetra-n-butyltin	10	15
98364036	Tetra-n-butyltin	10	15
98364036	n-Butyltin	10	62

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference

3. Initial Calibration

A six-point initial calibration was performed prior to each analytical batch The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD

4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent

5. Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this SDG

6 Surrogate Compound Recovery

Surrogate recovery goals for tri-n-propyltin were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment. Based on conversations with the laboratory an additional surrogate, tripentyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits
Tripropyltin	18 – 125%
Tripentyltin	28 – 122%

Surrogate compound percent recovery met quality control criteria for all samples

7 Laboratory Control Sample (LCS)

LCS recovery goals for butyltins were established in the project Sampling and Analysis Plan at 60 to 130 percent for sediment. Based on conversations with the laboratory, historical control chart limits of 8 to 161 percent for sediment were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following:

LCS	Analyte	% Recovery	QC Limit	Associated Samples
K981027-LCS	Di-n-butyltin	45	60-130 (P) 8-161 (L)	98364033 through 98364036
K981027-LCS	n-Butyltin	18	60-130 (P) 8-161 (L)	98364033 through 98364036

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were qualified as estimated (UJ) when LCS recoveries were outside project limits.

8. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan for sediment

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
Monbutyltin	10 - 120%

MS/MSD sample percent recoveries and relative percent differences (RPDs) met QC guidelines (P-project, L-laboratory), with the exception of the following:

Sample	Compound	% Recovery	QC Limits
K9806404-007MS (Batch QC)	Di-n-butyltin	16	30-120 (P) 8-144 (L)
K9806404-007DMS (Batch QC)	Di-n-butyltin	20	30-120 (P) 8-144 (L)

Additionally, MS and MSD recovery values for n-Butyltin were not calculated due to matrix interference. No action was taken based solely on MS/MSD data

9. Field Duplicate Analysis

No field duplicates were associated with this sample delivery group.

10. Sample Analysis

A cursory review of raw data was performed. Deliverables were complete. The case narrative indicated that the MS/MSD recovery of n-Butyltin for the Batch QC sample was not calculated because of matrix interference. The MRL required elevation due to this interference As the LCS recovery was within acceptance limits, no further action was taken. No other problems were noted.

11 Laboratory Contact

No laboratory contact was required.

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values

Data Qualifiers

U - The compound was analyzed for, but was not detected

UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met.

J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.

R - Quality control indicates that data are unusable (compound may or may not be present) Resampling and reanalysis are necessary for verification.

N - Presumptive evidence of presence of material (tentative identification).

I - Elevated reporting limit due to matrix interference.

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9806066
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/2/98
Sample Matrix:	Sediment	**Date Received:**	9/3/98

Butyltins

Sample Name	98364033	Units	ug/Kg (ppb)
Lab Code	K9806066-001	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	15	5	10/27/98	10/31/98	ND	15 UJ
Tri-n-butyltin	Method	Butyltins-GC	5	5	10/27/98	10/31/98	81	
Di-n-butyltin	Method	Butyltins-GC	5	5	10/27/98	10/31/98	20	J
n-Butyltin	Method	Butyltins-GC	5	5	10/27/98	10/31/98	23	J

D The MRL is elevated because of matrix interferences and because the sample required diluting

LGT 12/7/98

Approved By _____ Date 11-10-98

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9806066
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/2/98
Sample Matrix:	Sediment	**Date Received:**	9/3/98

Butyltins

Sample Name	98364034	Units	ug/Kg (ppb)
Lab Code	K9806066-002	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	15	5	10/27/98	10/31/98	ND	15UJ
Tri-n-butyltin	Method	Butyltins-GC	5	5	10/27/98	10/31/98	20	
Di-n-butyltin	Method	Butyltins-GC	5	5	10/27/98	10/31/98	6	J
n-Butyltin	Method	Butyltins-GC	5	5	10/27/98	10/31/98	ND	5UJ

D The MRL is elevated because of matrix interferences and because the sample required diluting

MQT 11/1/98

Approved By _____ Date 11-10-98

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9806066
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/2/98
Sample Matrix:	Sediment	**Date Received:**	9/3/98

Butyltins

Sample Name	98364035	Units	ug/Kg (ppb)
Lab Code	K9806066-003	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	15	5	10/27/98	11/1/98	5	J
Tri-n-butyltin	Method	Butyltins-GC	5	5	10/27/98	11/1/98	228	
Di-n-butyltin	Method	Butyltins-GC	5	5	10/27/98	11/1/98	52 J	
n-Butyltin	Method	Butyltins-GC	5	5	10/27/98	11/1/98	29 J	

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client:	Roy F Weston, Inc	**Service Request:**	K9806066
Project:	Duwamish River/4000-027-001-2019-38	**Date Collected:**	9/2/98
Sample Matrix:	Sediment	**Date Received:**	9/3/98

Butyltins

Sample Name	98364036	Units	ug/Kg (ppb)
Lab Code	K9806066-004	Basis	Dry
Test Notes	D		

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins-GC	15	5	10/27/98	11/1/98	ND	15UJ
Tri-n-butyltin	Method	Butyltins-GC	5	5	10/27/98	11/1/98	24	
Di-n-butyltin	Method	Butyltins-GC	5	5	10/27/98	11/1/98	8	J
n-Butyltin	Method	Butyltins-GC	62	5	10/27/98	11/1/98	ND	62UJ

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